



Technical Language Service

Translations From And Into Any Language

JAPANESE / ENGLISH TRANSLATION OF

Japanese Patent Application JP 3 - 146707 A

High-Toughness Rubber-Reinforcing Polyester Fiber

Your Ref: 081203-22

For: Eastman Chemical Company

9109 Eagle Hills Dr . Las Vegas NV 89134 . ph: 866-722-7990 (toll-free) . fax: 702-973-6009 . email: sales@tls-
translations.com

(19) Japanese Patent Office (JP)

(11) Unexamined Patent
Application (Kokai) No.

(12) Unexamined Patent Gazette (A)

3-146707

(51) Int. Cl. ⁵	Classification Symbols	Internal Office Registration Nos.	(43) Date of Publication: 21 June 1991
D 01 F 6/62	306 E	7199-4L	
C 08 J 5/04	CEQ	6639-4F	
D 01 F 6/62	302 C	7199-4L	

Request for Examination: Not yet submitted Number of Claims: 2 Total of 8 pages [in original]

- (54) Title of the Invention: **High-Toughness Rubber-Reinforcing Polyester Fiber**
- (21) Application No.: 1-287119
- (22) Date of Filing: 1 November 1989
- (72) Inventor: Futoshi SASAMOTO
c/o Mishima Plant, Toray Industries, Inc.
4845 Mishima-shi, Shizuoka Prefecture (no further details indicated)
- (72) Inventor: Takehiko MITSUYOSHI
c/o Mishima Plant, Toray Industries, Inc.
4845 Mishima-shi, Shizuoka Prefecture (no further details indicated)
- (72) Inventor: Shusei NAKAMURA
c/o Mishima Plant, Toray Industries, Inc.
4845 Mishima-shi, Shizuoka Prefecture (no further details indicated)
- (71) Applicant: Toray Industries, Inc.
2-1 Muromachi 2-chome, Nihonbashi, Chuo-ku, Tokyo

SPECIFICATION

1. Title of the Invention

High-Toughness Rubber-reinforcing Polyester Fiber

2. Claims

(1) A high-toughness rubber-reinforcing polyester fiber that has an amount of 5 ppm or less of antimony metal formed from the reduction of antimony compounds used as a polymerization catalyst, and that has the following properties.

- A. Carboxyl end group (COOH) content: $\leq 25 \text{ eg}^1/\text{ton}$
- B. Diethylene glycol (DEG) content: $\leq 1.3 \text{ wt } \%$
- C. Intrinsic viscosity (IV): ≥ 0.85
- D. Intermediate elongation + dry heat shrinkage (S): $\leq 12/0\%$
- E. Terminal modulus (M_t): $\leq 50 \text{ (g/d)}$

(2) The high-toughness rubber-reinforcing polyester fiber according to Claim 1, wherein the toughness ($T\sqrt{E}$) and dimensional stability (S) satisfy the expression $T\sqrt{E} \geq S + 19.5$.

3. Detailed Description of the Invention

(Field of Industrial Utilization)

The present invention relates to a high-toughness rubber-reinforcing polyester fiber. More specifically, the present invention relates to a rubber-reinforcing polyester fiber that has good dimensional stability, high strength, and exceptional durability.

(Prior Art)

Polyester fibers have exceptional mechanical properties, dimensional stability, and durability, and are widely used in industrial applications as well as in apparel. These fibers are extensively used in tire cords and other rubber material-reinforcing applications, as these characteristics are utilized therein. In the past, high-strength original yarns formed by stretching low-oriented unstretched yarns at a high ratio have been used in tire cord applications. In recent years, however, original yarns obtained by stretching relatively highly oriented unstretched yarns

¹ Translator's note: Possible misprint for "eq."

(so-called partially oriented yarns ("POY")) have come into use. This technique was developed in response to demands for better dimensional stability in the cords and for enhanced tire performance, and especially uniformity, despite a certain degree of strength being sacrificed.

(Problems That the Invention Is Intended to Solve)

However, tire cords having high strength and high toughness that still retain their dimensional stability have increasingly been required in recent years in order to improve tire performance. An example of a response to such requirements is disclosed in Japanese Patent Application (Kokai) Laid Open No. 60-88120, in which a POY is stretched at a low speed. However, the disclosed stretching technique provides only a minor range of improvement in the toughness (product of strength and elongation) of the fiber itself, meaning that the strength of tire cords in which the fiber is employed will not see any significant improvement, and the required level of strength and toughness will be insufficient. Reducing defects in the fiber is thought to be effective in improving toughness. In other words, various defects (foreign substances) present in tire cords are primarily caused by particles present in the polymer. These particles are often derived from polymerization catalysts, and it is accordingly of primary necessity to eliminate them.

Examples of catalysts used to manufacture rubber-reinforcing polyester fibers include manganese acetate, antimony trioxide, and phosphoric acid catalyst systems, which are disclosed in Japanese Examined Patent Application Publication (Kokoku) No. 37-5821; calcium acetate, antimony trioxide, and phosphorous acid systems, which are disclosed in Japanese Patent Application (Kokai) Laid Open No. 55-12871; and lithium acetate, antimony trioxide, and phosphorous acid systems, which are disclosed in Japanese Patent Application (Kokai) Laid Open No. 51-134789. Using these catalysts in polymerization will generate numerous catalyst-derived particles and create defects in the fiber. Accordingly, it will be impossible to satisfy the levels of high strength and toughness as required in the latest fibers.

The present inventors arrived at the present invention primarily by investigating polymers that could enable polyester fibers having good dimensional stability and exceptional strength and toughness to be obtained.

In other words, the present invention is a high-toughness rubber-reinforcing polyester fiber that has an amount of 5 ppm or less of antimony metal formed from the reduction of antimony compounds used as a polymerization catalyst, and that has the following properties.

- A. Carboxyl end group (COOH) content: ≤ 25 eg/ton
- B. Diethylene glycol (DEG) content: ≤ 1.3 wt %
- C. Intrinsic viscosity (IV): ≥ 0.85
- D. Intermediate elongation + dry heat shrinkage (S): $\leq 12/0\%$
- E. Terminal modulus (M_t): ≤ 50 (g/d)

As used herein, the term "polyester" refers to a polyester in which ethylene terephthalate is the principal repeating unit. To improve dimensional stability and strength, the polyester used is preferably a polyethylene terephthalate that does not have any added or copolymerized third components other than diethylene glycol byproducts, and it contains essentially no inorganic particles or other additives.

The present inventors conducted diligent investigations into factors that reduce the strength of the fibers, in order to increase their strength and toughness while retaining their good dimensional stability. As a result, the inventors identified the yarn defects, and in particular the catalyst-derived particles, that have prevented fibers from having increased strength. The inventors further discovered that among these particles, the metallic antimony formed from the reduction of antimony compounds used as a polymerization catalyst imparts a profoundly adverse effect on strength and toughness. The inventors accordingly conducted research on how the amount of metallic antimony relates to the performance of the resulting polyester fiber; and particularly to the durability of fiber toughness, and discovered that toughness could be improved to a significantly greater extent than experienced in the past if the amount of the antimony metal was established at 5 ppm or less.

From the above perspective, the effect was found to be even more pronounced when the amount of antimony metal was established at 3 ppm or less. Although not using an antimony compound as a polymerization catalyst was also considered for reducing the antimony metal content in the fiber, the absence of an antimony compound will cause the dimensional stability of the fiber to decline, and other properties of the polymer (e.g., the content of carboxyl end groups

or DEG) to deteriorate under commercial manufacturing conditions. Consequently, it is necessary for the amount of antimony metal formed from reduction when an antimony compound is used as a polymerization catalyst to be 5 ppm or less, in order to ensure the dimensional stability and durability required of rubber-reinforcing polyester fibers and to improve their toughness. From the above perspective, the amount of antimony compound used as a polymerization catalyst is preferably 60 to 150 ppm as antimony metal. Antimony trioxide and antimony pentoxide are preferred examples of antimony compounds used as polymerization catalysts.

The carboxyl end group (COOH) content in the polyester fiber of the present invention must be 25 eg/ton or less. If the COOH content exceeds 25 eg/ton, the fiber will quickly deteriorate within the rubber and prove insufficiently durable as a rubber-reinforcing material.

From the above perspective, the COOH group content is preferably 21 eg/ton or less.

The diethylene glycol (DEG) content in the polyester fiber of the present invention must be 1.3 wt % or less. If the DEG content exceeds 1.3 wt %, the dimensional stability of the fiber will be poor, and its durability in the rubber will be reduced. From the above perspective, the DEG content is preferably 1.1 wt % or less.

The intrinsic viscosity (IV) of the polyester fiber of the present invention must be 0.85 or greater. If the IV is less than 0.85, the fatigue resistance of the fiber will be poor, rendering the fiber unusable as a rubber-reinforcing material. From the above perspective, the IV is preferably 0.90 or greater. However, the IV is preferably 1.3 or less in terms of fiber spinnability and other operational aspects, such as stability.

The dimensional stability (intermediate elongation + dry heat shrinkage) of the polyester fiber of the present invention must be 12% or less. If the dimensional stability exceeds 12%, poor dimensional stability of the cord will result when used in molding tires, and the uniformity of the tire will be lowered. From the above perspective, the dimensional stability is preferably 10% or less, and more preferably 8% or less.

The terminal modulus of the polyester fiber of the present invention must be 50% or less. If the terminal modulus exceeds 50 g/d, the fiber will have low tenacity when twisted, rendering

any increase the tire cord strength impossible, even if the strength of the original yarn has been increased. From the above perspective, the terminal modulus is preferably 35 g/d or less.

As has been described in the foregoing, defects in the fiber can be dramatically reduced by strictly controlling the amount of antimony metal formed from reducing antimony compounds used as a polymerization catalyst, and a polymer with a satisfactory COOH group and DEG content will be obtained. It is only when such a polymer is used that polyester fibers that exhibit unprecedentedly high levels of performance can be manufactured.

Rubber-reinforcing materials such as tire cords require improved toughness along with good dimensional stability; i.e., a consistently low intermediate elongation + dry heat shrinkage value.

The present inventors also conducted diligent investigations into such requirements, and as a result discovered that strictly controlling the amount, type, and addition method of the phosphorus compound used in polymerization is crucial in meeting the aforementioned requirements. Phosphorus compounds are generally used to improve the heat resistance of the polymer; however, as a result of their research, the present inventors quite unexpectedly made the novel discovery that the phosphorus compounds affect the relation between the dimensional stability and toughness of the fiber, as has been described in the foregoing.

As a result of their research, the present inventors discovered that it is preferable for phosphoric acid to be used as the phosphorus compound, for the residual amount of this compound to be 10 to 40 ppm in terms of elemental phosphorus, and for the phosphorus compound to be added at the start of polycondensation. Controlling the phosphorus compound in such a manner will enable the toughness of the fiber to be raised at the same level of dimensional stability. In other words, the aforementioned conditions are preferred because higher fiber toughness can be obtained even when the dimensional stability is the same, such as when the following equation is satisfied:

Strength-elongation product ($T\sqrt{E}$) \geq (intermediate elongation + dry heat shrinkage) + 19.5.

(where T is strength (g/d) and E is elongation (%)).

Although the cause behind the effect of the phosphorus compounds remains uncertain, the present inventors have presumed that the formation of the fiber structure during spinning is controlled by the viscosity-enhancing effect of phosphoric acid or other trifunctional phosphorus compounds.

Tire cords that have significantly higher fatigue resistance than seen conventionally can be manufactured by using an original yarn that satisfies the equation $T\sqrt{E} \geq S + 19.5$ (where S = intermediate elongation + dry heat shrinkage), as has been described in the foregoing. In other words, the higher the toughness of the original yarn, the better will be the fatigue properties of the tire in general. However, the correlation between such toughness and fatigue properties will vary with the dimensional stability (S), and a lower dimensional stability will yield better fatigue properties at the same toughness. In other words, the present inventors discovered that if $T\sqrt{E} \geq S + 19.5$ is satisfied for toughness versus dimensional stability (S), the fatigue resistance will be significantly higher than experienced in the past.

From the above perspective, $T\sqrt{E} \geq S + 22.0$ is more preferable. It is only with such a relationship between $T\sqrt{E}$ and S that an operation for improving dimensional stability can be performed at the polymer level, and the defects in the fiber caused by the antimony metal can be significantly reduced in accordance with the present application.

According to the present invention, the following can be cited as examples of methods for controlling the production of metallic antimony, when an antimony compound is used as a polymerization catalyst.

- (1) Increase the surface-area-to-volume ratio in the molten polymer during polymerization, and reduce the polymerization time. In this method, the polymerization time is preferably less than one hour, and more preferably less than 30 min.
- (2) Keep the amount of the antimony compound to 130 ppm or less in terms of antimony while also using another polymerization catalyst (preferably an organotitanium compound or a tin compound) during polymerization.

(3) Lower the final polymerization temperature. In this method, the temperature should be lowered to 290°C or less, and more preferably to 285°C or less.

The fiber of the present invention can be obtained, for example, by means of the following method, but production of the fiber shall not be limited to this method alone.

A polycondensation reaction is conducted while using an antimony compound (130 ppm or less in terms of antimony) and a titanium compound as polymerization catalysts. In the reaction, it is preferable to use phosphoric acid as the phosphorus compound, and to add the phosphoric acid at the start of polycondensation, before the antimony compound and titanium compound are added. Polyethylene terephthalate chips (IV of 0.65 or greater; COOH group content ≤ 26 eg/ton; DEG content ≤ 1.3 wt %) will be obtained by suitably selecting the amounts of the components introduced, the temperature of polymerization, and time of polymerization.

The resulting chips will yield polyethylene terephthalate (IV or 1.0 or greater, and COOH group content ≤ 16 eg/ton) when subjected to solid-phase polymerization in accordance with a known method.

The resulting solid-phase polymerized chips are slowly cooled in the heating zone after being melt-spun in accordance with a known method, after which the yarn is taken up while being cooled and solidified in a chimney air current. The piping and packing components in the spinning machine are preferably plated with chromium so as to inhibit precipitation and reduction of the antimony metal. Nonwoven fabric with metal wire (S, sodium), fashioned to an absolute filtering diameter not larger than 30 μm , is preferably used for filtration. It is also preferable to minimize the amount of dust in the elements used in solid-phase polymerization and in the nitrogen used in the spinning machine, and to reduce the dust content by filtering the air used for the chimney air current. The quantity of foreign substances produced in the yarn in the aforementioned yarn-forming method will be kept to a level of 800/mg or less, and preferably 500/mg or less. This method is preferred because improved toughness and durability are obtained at these levels.

High-orientation spinning is performed, with the yarn discharged from the spinneret being taken up at a rate of 1000 m/min, and preferably 2000 m/min. The unstretched yarn is

spun, and then stretched on hot rollers for heat setting at a temperature of 220°C or higher, either directly thereafter or after having being wound once. A terminal modulus of 50 g/d or less can be obtained by suitably selecting the draw ratio and relaxation rate in this process.

(Working Examples)

The present invention shall be described in further detail below using working examples.

The properties of the working examples were measured according to the following methods.

- A. The amounts of metal (e.g., antimony and phosphorus) in the polymer and fiber were determined by X-ray fluorescence analysis.
- B. Carboxyl end group ([COOH]) content
A 0.5 g sample was dissolved in 10 mL *o*-cresol, and cooled after having completely dissolved. 3 mL chloroform was then added into the resulting solution, and potentiometric titration was performed using a methanol solution of NaOH.
- C. DEG content
Gas chromatography was used after the sample had been subjected to alkalinolysis.
- D. Degree of elongation, intermediate elongation, and terminal modulus
A Tensilon tensile tester (Toyo Baldwin) was used to determine the S-S curve at a sample length of 25 cm and a take-up rate of 30 cm/min, and to compute the degree of elongation.

The intermediate elongation was determined from the same S-S curve by reading the elongation corresponding to a strength of 4.5 g/d. The terminal modulus was determined by means of obtaining the difference between the breaking stress and the stress taken at the point where the elongation at break had been reduced by 2.4%, and dividing the difference by 2.4×10^{-2} .

E. Dry heat shrinkage (ΔS_d)

A sample in the form of a hank was left for 24 hours or more in an air-conditioned chamber in conditions of 20°C and 65% RH, after which the length of the sample (l_0) was measured under a load that corresponded to 0.1 g/d of the sample. The sample was then left for 15 min in an oven that had been heated to 150°C, in a non-tensioned state. The sample was then removed from the oven and again left for four hours in the aforementioned air-conditioned chamber, after which its length (l_1) was again measured under the aforementioned load. The dry heat shrinkage (ΔS_d) was calculated according to the following equation:

$$\Delta S_d = (l_0 - l_1) / l_0 \times 100 (\%)$$

F. Number of foreign substances in yarn

The sample was separated into single filaments (length: 6 cm) and pulled completely taut on a glass slide to be used for sampling. The number of foreign substances in the yarns was counted by the phase contrast method using an optical microscope (Olympus Optical Co., Ltd.) to scan the sample at a 200-times magnification. The measurements were performed 5 times ($N = 5$) to determine the mean value X (substances per 6 cm), which was converted into the number of foreign substances per milligram.

G. Intrinsic viscosity (IV)

0.8 g of sample was dissolved in 10 mL *o*-chlorophenol ("OCP" hereunder) at a temperature of 25°C, after which an Ostwald viscometer was used to determine the relative viscosity ($\eta\gamma$) based on the following equation, and subsequently the IV.

$$\eta\gamma = \eta / \eta_0 = t \times d / t_0 \times d_0$$

$$IV = 0.0242\eta\gamma + 0.2634$$

η	:	viscosity of polymer solution
η_0	:	viscosity of solvent
t	:	solvent falling time (sec)

d	:	density of solution (g/cm^3)
t_0	:	OCP falling time (sec)
d_0	:	density of OCP (g/cm^3)

H. Amount of antimony metal

40 g of polymer was dissolved in 500 mL *o*-chlorophenol (OCP), and the resulting solution was centrifuged ($12,000 \text{ rpm} \times 2 \text{ hr}$). The separated products were washed and dried. The spectra of the centrifugally settled particles were measured using an X-ray diffraction device, and used to determine the amount of metallic antimony.

I. Heat resistance in rubber

Cords were embedded in rubber and vulcanized for 6 hr at 150°C . The heat resistance was evaluated according to the tenacity rate. The tenacity rate was graded as "1" (70% or better); "2" (60 to 70%); and "3" (less than 60%).

J. Fatigue resistance (GY fatigue life)

The tube burst time was determined according to ASTM – D885, with an internal tube pressure of 3.5 kg/cm^2 , a rate of rotation of 850 rpm, and a tube angle of 90° . The results are shown as follows.

- "1" At least 30% better than a conventional product (commercial tire cord model 1000-240-703M; Toray Industries, Inc.)
- "2" 10 to 30% better than the conventional product.
- "3" The same level as the conventional product

(Working Example 1)

(Polymer A)

0.035 parts manganese acetate tetrahydrate was added to 100 parts dimethyl terephthalate and 50.2 parts ethylene glycol, and the resulting mixture was subjected to transesterification according to a normal method. 0.015 parts phosphoric acid was added to the resulting product, after which 0.032 parts antimony trioxide was further added, and the resulting mixture was

subjected to a polycondensation reaction for three hours and ten minutes (final polymerization temperature: 285°C).

The IV of the resulting polymer was 0.72, the COOH group content was 14 eg/ton, and the DEG content was 0.58 wt %. 10 ppm antimony metal was present in Polymer A.

(Polymer B)

Thin-film polymerization was conducted using the same catalyst composition as in Polymer A, with a film thickness of 0.8 mm being adopted (time of polymerization: 57 min).

The IV of the resulting polymer was 0.68; the COOH group content was 10 eg/ton, and the DEG content was 0.5 wt %. 0.3 ppm antimony metal was present in Polymer B.

(Polymer C)

A combined polymerization catalyst system of antimony trioxide and tetrabutyl titanate was subjected to a polycondensation reaction under the same conditions as in the manufacture of Polymer A, with the amount of added catalyst being varied as shown in Table 1. The properties of the resulting polymer are all shown in Table 1.

Table 1

	Amount of catalyst added		COOH	DEG	Sb metal (ppm)
	Sb ₂ O ₃	Ti(OC ₄ H ₉) ₄			
A	0.032	0	14	0.58	10
B	0.032	0	10	0.68	0.3
C-1	0.020	0.015	16	0.62	6.4
C-2	0.015	0.025	18	0.70	4.5
C-3	0.0125	0.025	18	0.70	1.0
C-4	0.010	0.03	16	0.65	0.6

As is evident from the data in Table 1, the amount of antimony metal can be reduced either by means of the method involving the combined use of an antimony catalyst and an organic titanium catalyst, or by the thin-film polymerization method, in which the polymerization time is small.

Next, Polymers A and C-1 to C-4 shown in Table 1 were melt-spun and stretched to form polyester fibers, according to the method below.

(Spinning method)

The aforementioned polymers were pre-dried for 5 hrs at 160°C, and then subjected to solid-phase polymerization at a temperature of 225°C. The resulting solid-phase polymer chips had intrinsic viscosities of 1.35 to 1.40. The chips were spun from a spinneret having round orifices with a diameter of 0.6 mm at a spinning temperature of 295°C using an extruder-type spinning machine, while being filtered using a nonwoven metal fabric having an absolute filtration diameter of 15 μ m as a filter. The discharged yarns were cooled in a chimney after having been slowly cooled in a heating cylinder that had a length of 25 cm and had been heated to a temperature of 300°C. The yarns were taken up at a rate of 2,000 m/min. The resulting unstretched yarns were stretched and subjected to a heat treatment under conditions corresponding to a stretching temperature of 90°C, a heat treatment temperature of 240°C, and a relaxation rate of 1.5%, to yield stretched yarns. The resulting stretched yarns had intrinsic viscosities of 0.98 to 1.03, COOH group contents of 12 to 18 eg/ton, and DEG contents of 0.58 to 0.7%.

Next, preliminary twists of 49 T/cm were applied to the stretched yarns in the S direction, and final twists of 49 T/cm were then applied in the Z direction, to obtain green cords.

These cords were dipped in an adhesive using a Computreater (C. A. Litzler Co., Inc.) to form treated tire cords. The treatment conditions were: 160°C for a length fixing treatment in the drying unit; 240°C for a tension treatment in the heat treatment unit; and 240°C for a relaxation treatment in the post-treatment unit. The intermediate elongation of the treated cords was brought to 3 to 4% by adjusting the tensioning and relaxation rates. The properties of the original yarns, green cords, and treated cords are shown in Table 2.

Table 2

No.	Sb metal (ppm)	Foreign substances in yarn (mg)	Strength (g/d)	Elongation (%)	Intermediate elongation in original yarn (%)	Dry shrinkage (%)	Terminal modulus	$\sqrt{T/E}$	Strength of treated cord (g/d)	Fatigue resistance
A	10	4,280	7.7	12.9	6.8	2.4	32	27.7	6.2	4 to 3 ²
C-1	6.4	2,500	7.9	12.8	6.9	2.3	40	28.3	6.6	3
C-2	4.5	1,300	8.5	12.6	6.9	2.4	35	30.2	6.8	2
C-3	1.0	520	9.2	13.3	6.9	2.2	32	33.6	7.1	1
C-4	0.6	300	9.3	13.8	6.8	2.4	31	34.5	7.3	1
C-3'	1.0	555	9.6	10.8	6.5	2.9	60	31.2	6.6	2

As is evident from the data in Table 2, there were numerous defects in the yarns (as shown in the "foreign substances in yarn" column) in the fibers obtained from A and C-1, where the amount of Sb metal exceeded 5 ppm, and these fibers had low toughness. However, the amount of foreign substances had decreased and toughness had significantly improved in the fibers obtained from C-2 to C-4, because controlling the amount of antimony catalyst kept the amount of Sb metal to 5 ppm or less. This effect was especially pronounced in C-3 and C-4, where the amount of Sb metal was 3 ppm or less. The strength of the treated cord was found to have decreased in C-3', which had been subjected to the same conditions as C-3 except for an increased draw ratio, which increased the terminal modulus.

Examining the levels of fatigue resistance revealed that C-2 to C-4 and C-3' performed better than conventional articles, as they were the only polymers obtained whose $T\sqrt{E}$ in the original yarn and dimensional stability (S) satisfied $T\sqrt{E} \geq S + 19.5$. Especially good results were produced in C-2 to C-4, wherein $T\sqrt{E} \geq S + 22.0$.

(Working Example 2)

Polymer C-3 was subjected to solid-phase polymerization under various conditions and spun to yield the fibers in Table 3.

² Translators' note: The category of "4" does not appear in the explanation of the gradations under the fatigue resistance description that appears earlier in the document, although it appears here in the table.

Table 3

No.	Take-up rate (m/min)	COOH	IV	Strength (g/d)	Elongation (%)	Intermediate elongation of original yarn (%)	Dry shrinkage (%)	Dimensional stability (S)	Heat-resistance in rubber	Tire uniformity	Fatigue resistance
2-1	500	15	0.98	10.8	10.7	3.5	10.2	13.7	1	3	2
2-2	1,000	15	-	10.2	10.9	3.9	7.9	11.8	1	2	2
2-3*	2,000	15	-	9.2	13.3	6.9	2.2	9.1	1	2	1
2-4	2,000	22	0.95	9.2	12.9	6.9	2.3	9.2	2	2	1
2-5	2,000	27	0.90	9.1	13.1	6.8	2.4	9.2	3	2	1
2-6	2,000	20	0.74	8.5	13.0	6.6	1.5	8.1	2	2	3

* same as C-3

A reduced take-up rate was used in No. 2-1 in Table 3, which caused the dimensional stability to exceed 12.0% and tire uniformity to decline. The heat resistance in No. 2-5 was poor, as the carboxyl end group content exceeded 25 eg/ton. No. 2-6 exhibited poor fatigue resistance, due to its IV of less than 0.8.

(Working Example 3)

Diethylene glycol was added during polymerization using the same method as C-3 to obtain polymers having DEG contents as shown in Table 4. Tire cords were obtained by means of spinning, stretching, and after-processing these polymers under the same conditions as in Working Example 1. The heat resistance of these cords was evaluated, and the results are also displayed in Table 4.

Table 4

No.	Amount of DEG	Heat resistance
3-1	1.5	3
3-2	1.2	2
3-3 (same as C-3)	0.7	1

As shown in Table 4, the heat resistance was unsatisfactory because the DEG content had exceeded 1.3%.

(Effect of the Invention)

As has been described in the foregoing, it is only by strictly controlling the catalyst composition and by keeping the amount of antimony metal to a minimum that rubber-reinforcing original yarns with high toughness, dimensional stability, heat resistance, and durability can be obtained. The original yarns are particularly useful for tire cords, which can be used to produce more-lightweight tires since they will have fewer ends and plies than conventional tires. The good dimensional stability of the fiber will allow tires with good uniformity to be obtained.

Applicant: Toray Industries, Inc.